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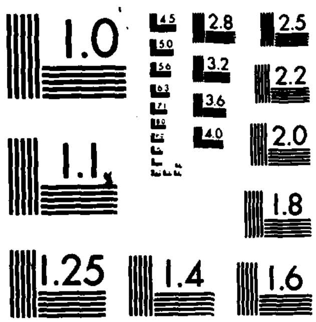

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REPORT SD-TR-83-92

(12)

AD A138911

Pulsed D_2-F_2 Chain-Laser Damage to Coated Window and Mirror Components

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15 December 1983

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Prepared for

SPACE DIVISION
AIR FORCE SYSTEMS COMMAND
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Los Angeles, California 90009

This report was submitted by The Aerospace Corporation, El Segundo, CA 90245, under Contract No. F04701-83-C-0084 with the Space Division, P.O. Box 92960, Worldway Postal Center, Los Angeles, CA 90009. It was reviewed and approved for The Aerospace Corporation by W. P. Thompson, Director, Aerophysics Laboratory. Captain D. H. Olsen, SD/YNS, was the project officer for the Mission Oriented Investigation and Experimentation (MOIE) Program.

This report has been reviewed by the Public Affairs Office (PAS) and is releasable to the National Technical Information Service (NTIS). At NTIS, it will be available to the general public, including foreign nationals.

This technical report has been reviewed and is approved for publication. Publication of this report does not constitute Air Force approval of the report's findings or conclusions. It is published only for the exchange and stimulation of ideas.

Capt. Douglas H. Olsen	Joseph Hess
Douglas H. Olsen, Captain, USAF Project Officer	Joseph Hess, GM-15, Director West Coast Office, Air Force Space Technology Center

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REPORT DOCUMENTATION PAGE		READ INSTRUCTIONS BEFORE COMPLETING FORM
1. REPORT NUMBER SD-TR-83-92	2. GOVT ACCESSION NO. A138911	3. RECIPIENT'S CATALOG NUMBER
4. TITLE (and Subtitle) PULSED D_2-F_2 CHAIN-LASER DAMAGE TO COATED WINDOW AND MIRROR COMPONENTS		5. TYPE OF REPORT & PERIOD COVERED 6. PERFORMING ORG. REPORT NUMBER TR-0084(4930-01)-2
7. AUTHOR(s) S. T. Amimoto, J. S. Whittier, A. Whittaker, A. Chase, R. Hofland, Jr., and M. Bass		8. CONTRACT OR GRANT NUMBER(s) F04701-83-C-0084
9. PERFORMING ORGANIZATION NAME AND ADDRESS The Aerospace Corporation El Segundo, Calif. 90245		10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS
11. CONTROLLING OFFICE NAME AND ADDRESS Space Division Los Angeles Air Force Station Los Angeles, Calif. 90009		12. REPORT DATE 15 December 1983 13. NUMBER OF PAGES 20
14. MONITORING AGENCY NAME & ADDRESS(if different from Controlling Office)		15. SECURITY CLASS. (of this report) Unclassified 15a. DECLASSIFICATION/DOWNGRADING SCHEDULE

16. DISTRIBUTION STATEMENT (of this Report)

Approved for public release; distribution unlimited

17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)

18. SUPPLEMENTARY NOTES

19. KEY WORDS (Continue on reverse side if necessary and identify by block number)

Acid resistance	DF-chain laser
Adhesion strength	Laser damage
Carbyne (carbon) coatings	Laser mirrors
Coating absorption,	Laser windows

20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

Large-spot laser damage thresholds were measured for bowl-feed-polished CaF_2 and sapphire windows (bare and antireflection-coated) and for highly polished copper mirrors (bare and carbyne-coated) at DF chain-laser wavelengths (3.58-4.78 μm). The chain reaction between F_2 and D_2 was initiated by a magnetically confined electron beam, producing DF-laser outputs of 10 to 20 J in pulses of 0.6 to 0.9 μsec (FWHM) duration. Energy extracted from a transmission-coupled unstable resonator was focused by means of a

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micrometers microseconds

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19. KEY WORDS (Continued)

20. ABSTRACT (Continued)

CaF_2 lens. A soft-aperture technique was employed to suppress effects of Fresnel diffraction so that uniform (top-hat) intensity profiles were obtained along the focusing beam. With this laser system, commercially available antireflection-coated CaF_2 and Al_2O_3 samples were tested and found to have damage thresholds from 17 to 28 J/cm^2. Significantly larger damage thresholds were observed for uncoated, polished samples of Al_2O_3, but damage resistance of uncoated polished CaF_2 was found to equal that of the best antireflection-coated CaF_2 samples. A highly polished copper mirror had the highest damage threshold of all the materials tested, i.e., 58 J/cm^2. Carbyne films of diamond-like hardness, a type of carbon coating, were applied to polished copper mirrors and bowl-feed-polished CaF_2 surfaces. The carbyne coatings prepared in this work contained numerous carbon-bearing particles that were easily damaged (~10 J/cm^2). However, regions of the irradiated carbyne film that were free of carbon particles withstood high laser fluences (25 J/cm^2), indicating that improvements in carbyne film preparation would yield protective coatings with high damage resistance at DF wavelengths.

CONTENTS

FIGURES

I. INTRODUCTION

The performance of optical components can significantly affect the size, weight, and efficiency of high-power laser systems. Component failure in pulsed DF chain-reaction lasers presently imposes design limitations on laser gain length and aperture size. Unique challenges associated with component development for DF chain lasers are the laser's broadband spectral output of 3.58 to 4.78 μm, the requirement for operation in the presence of a corrosive F_2/DF environment, and the need to withstand high impact loadings associated with combustion overpressures of from 6 to 8 atmospheres. Data on the failure of DF laser optical components are restricted primarily to small-energy pulses and, hence, to small spot sizes of the order of hundreds of micrometers.[1,2] The scaling of these results to large spot sizes of practical importance is known to be unreliable. Damage thresholds of 52 J/cm^2 were recently reported for $ThF(ZnS)_3$-coated mirrors[3] using beam spots up to 1 cm in diameter.[4] Such excellent damage resistance has, however, been observed only in benign (air) environments. Similar coatings on internal mirrors and windows mounted in repetitively pulsed systems have repeatedly failed after several shots at incident fluences of several joules per square centimeter. Damage was by acid etching and by blowoff induced by laser irradiation.

Coating failure in pulsed chain-laser systems has prompted us to examine the suitability of carbyne coatings as hard protective films on pulsed DF-laser optical components. Chaoite, the carbyne form of interest, has been produced at The Aerospace Corporation by means of quench cooling of carbon gas.[5,6] Early studies of chaoite films revealed several interesting properties: (1) diamond-like hardness (greater than B_4C), (2) chemical resistance to acids, bases, and organic solvents, (3) good adhesion to copper, platinum, glass, silicon, germanium, sapphire, and other materials, and (4) low absorption in the 2- to 40-μm range. In view of these attractive properties, we undertook a brief program to apply carbyne films to candidate DF-laser window and mirror materials for evaluation with regard to adhesion strength, resistance to HF/DF attack, abrasion resistance, optical absorption, and laser

damage threshold. During the two-week study of laser damage resistance, we also measured damage thresholds for selected commercially available coatings applied to both transparent and reflective optics for purposes of comparison with chaoite film performance. The resulting data show that the best chaoite films have high damage resistance, hardness, and resistance to acid attack, and may be attractive for use on pulsed-DF-laser optical components if coatings can be developed that are free of carbon particles.

II. EXPERIMENTAL TECHNIQUE

A diagram of the laser damage apparatus employed in the present study is given in Fig. 1. A magnetically confined electron beam was used to initiate the pulsed chain-reaction DF laser.[7] Mixtures containing 20% F_2- 8% D_2 by volume were irradiated for periods of 0.1 to 1 μsec at current densities of 20 A/cm^2 to accomplish laser initiation. Nominal laser energies of 10 to 20 J in 0.6 to 0.9 μsec (FWHM) pulses were delivered at cavity pressures of 800 Torr. Energy was extracted from the gain medium by means of a transmission-coupled half-symmetric unstable resonator and then collimated using a CaF_2 lens of 8-m focal length. Laser windows were uncoated, 1.25-cm-thick CaF_2 crystals that were tilted with respect to the optical axis. A beam splitter at nearly normal incidence to the laser beam diverted about 6% of the total pulse energy into a 9-cm ballistic thermopile. Emission time history of the laser was monitored with a gold-doped germanium detector. In a previous study, the D_2-F_2 laser spectral output was measured and found to consist of up to 69 lines operating between 3.58 and 4.78 μm.[8]

The remaining energy in the pulsed DF-laser beam was focused by means of a CaF_2 lens of 45-cm focal length. The lens was translated along the direction of the beam to vary the fluence incident upon the optical test sample. A fluence range of 5 to 70 J/cm^2 could be encompassed by this technique. To obtain uniform beam spatial distributions along the focusing beam, an intracavity soft-aperture filter was employed (Fig. 1). This filter suppressed effects of Fresnel diffraction during focusing. The spatial distribution of the focusing beam was determined from burn patterns on calibrated witness film (Fig. 2). As illustrated in Fig. 2, a uniform (top-hat) spatial fluence distribution was obtained by use of the soft-aperture filter.

Laser damage measurements were carried out on a variety of transparent and reflective optics in the present work (Table I). Antireflection (ar)-coated windows of Al_2O_3 and CaF_2 were obtained from Laser Power Optics (LPO) and CVI Laser Corporation. Coatings on the Al_2O_3 samples were ZnS/ThF_4 and $TiO_2/Al_2O_3/MgF_2$; the ar coating on the CaF_2 sample was PbF_2/ThF_4. Details of

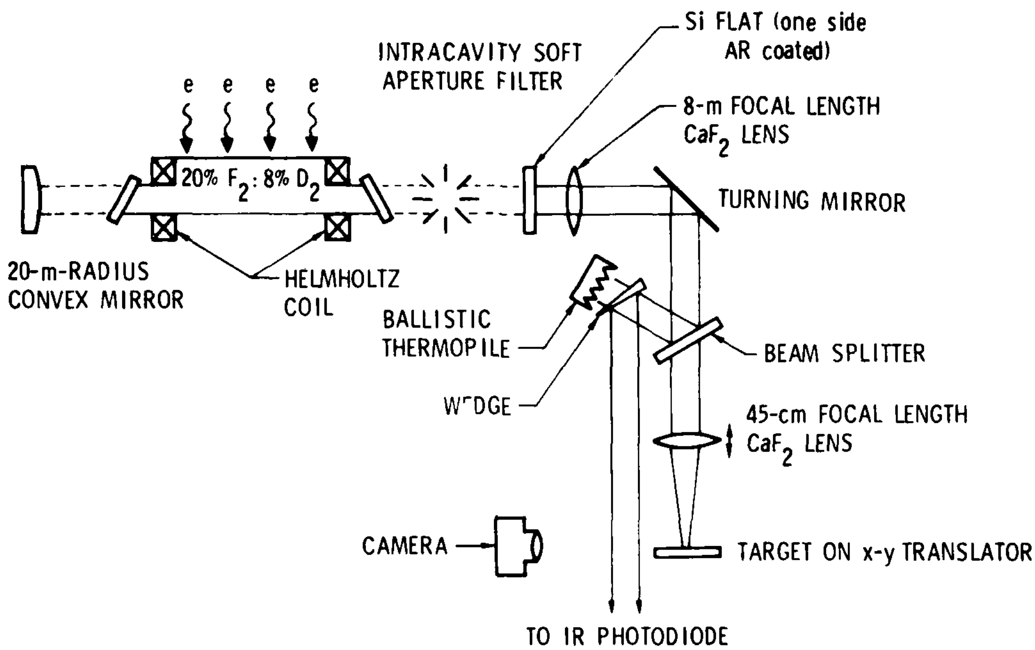

Fig. 1. Laser Damage Apparatus Layout

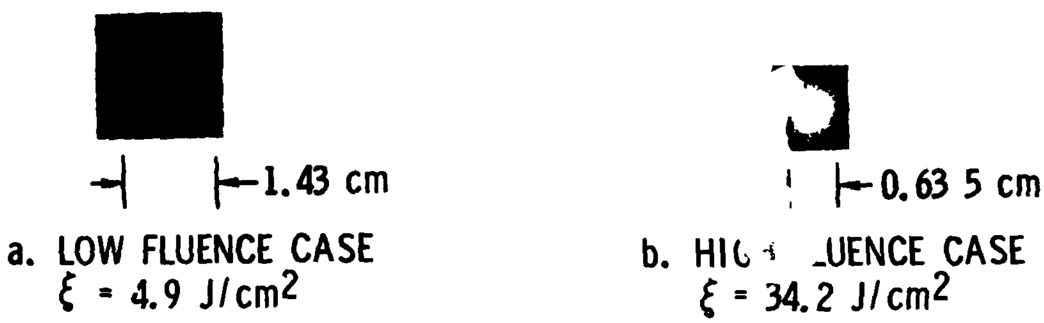

Fig. 2. Calibrated Film Burns Showing Typical DF-Beam Spatial Fluence Distributions

Table I. DF-Laser-Damage Summary

Substrate	Substrate Thickness, mm	Coating (Vendor)	Type of Damage	Fluence, J/cm^2
Al_2O_3	6.25	None	Isolated spot on exit surface	25
			No entrance damage	<54
	3.12	$TiO_2/Al_2O_3/MgF_2$ (CVI)	Isolated spot on exit surface coating	28
			Damage to entrance surface coating	38
	3.12	ZnS/ThF_4 (LPO)	Isolated damage on entrance and exit coating	17
CaF_2	5	None	Exit surface and bulk damage at lens focus	21
			Entrance surface and bulk damage at lens focus	27
	5	PbF_2/ThF_4 (LPO)	Exit coating damage	21
			Isolated spot on front	27
	5	Carbyne (Aerospace)	Small spot on entrance coating	14
			No visible damage	25
Cu	-	None (Spawr)	Slight surface damage	58
	-	Carbyne (Aerospace)	Severe coating failure	9.5

the coating designs are available from the vendors.[9,10] Bowl-feed-polished samples of Al_2O_3 and CaF_2 were also tested as a standard against which coated window performance could be assessed. Two mirrors were tested in our study: an uncoated laboratory-grade copper mirror (Spawr) and a carbyne-coated copper mirror. The performance of carbyne-coated CaF_2 windows was also evaluated in our work.

Transparent carbon films for use in the present study were produced by quenching carbon gas on selected substrates, including transparent and reflective optical elements.[11] A schematic diagram of the equipment that was used to produce exploratory carbyne films is shown in Fig. 3. The sample to be coated and a piece of pure carbon were located near the center of the coating pressure vessel. The optical sample could be heated to 180°C and was discharge cleaned prior to coating application. The pure carbon source was positioned in a carbon mandrel that was held in the chuck of a spinner mounting. The spinning carbon target was heated by means of a focused 1.5 kW CO_2 laser beam that entered the chamber through a NaCl window. During laser irradiation, the hot carbon rod was surrounded by a cloud of carbon gas that impinged on the surface of the spinning optical sample. The temperature of the solid carbon source was measured by an optical pyrometer of fast response time (~0.1 sec). The pyrometer was located, as shown in Fig. 3, with its line of sight at 30 deg to the laser beam axis. Careful temperature control of the carbon source was crucial for production of the desired carbyne film. Chamber pressure was measured by a Baratron gauge. Instability in the output of the CO_2 laser resulted in difficulties in the preparation of uniform, reproducible coatings. In view of the unsophisticated nature of the coating apparatus, it is believed that improvement in the quality and reproducibility of carbyne coatings should be readily achievable.

Fifty laser shots were performed on the 10 samples that were available for our damage study. Approximately one full day of testing was required to determine the damage threshold of an optical component. During testing, the sample was rotated after each exposure so that a fresh area was irradiated. The large spot sizes used in our study implied that only a few exposures could

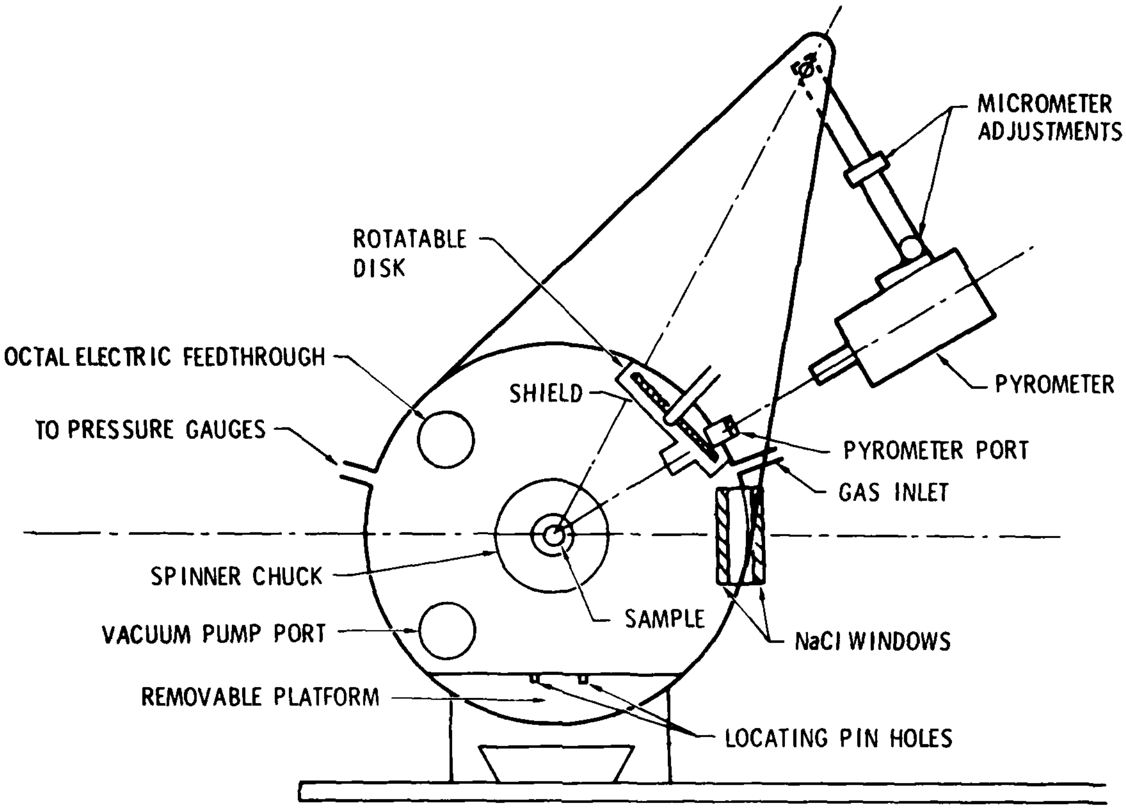

Fig. 3. Diagram of Equipment Used to Apply Carbyne Films to Optical Components

be performed on each optical sample. Exposure began below 10 J/cm^2 and was increased in steps of 50 to 100% until small-scale and, finally, catastrophic damage was observed. Component damage was determined by post-irradiation inspection with a 10X microscope, using strong illumination.

III. RESULTS AND DISCUSSION

Selected properties of carbyne films were briefly examined before measurement of laser damage resistance. Absorption coefficients in the infrared were found to increase from approximately 2×10^2 cm^{-1} at 10 μm to approximately 2×10^3 cm^{-1} at 1.0 μm. Index of refraction averaged about 2.1; one film, however, gave a rather low index value of 1.7. Absorption peaks at 3.1, 5.9, 6.2, 7.3, and 8.5 μm strongly indicated that the present carbyne films were contaminated with pump oil. Contamination was possible because no precautions were taken to prevent backstreaming of pump oil during the carbyne coating process. Because pump oil would be expected to increase absorption in the 3.6 to 4.8 μm bandpass, its presence probably would have reduced the DF damage resistance of the carbyne-coated samples that were tested in this work.

An ion-microprobe mass analyzer (IMMA) was used to obtain unequivocal identification of carbyne as the film that was produced. Although two different carbon negative-ion spectra have been observed using the IMMA, carbon films used in this study gave spectra that stopped at C_4. Unfortunately, the IMMA could not identify the particular carbyne form that was produced in our work.

Abrasion tests were also performed on the carbyne films. The results revealed that the films were more abrasion resistant than their substrates (Ni, Al, Cu, CaF_2); they were determined, however, to be less resistant than fused quartz. Adhesion strength of the carbyne films varied over the range from 57 to 652 kg/cm^2. The diffusion of vacuum pump oil onto the substrates could have accounted for these low values as well as the wide variation in adhesion strength.

Chemical resistance of the carbyne films was evaluated using concentrated HF acid. The films were unaffected by the acid. Penetration of the films by way of imperfections was observed, however. One continuous film was tested that was not penetrated during the time required for the droplet of acid to evaporate (about 30 minutes).

The laser damage threshold measurements performed during the present study are summarized in Table I. Included in the table are component substrate material, substrate thickness, component coating (when present), coating vendor, type of damage observed, and incident laser fluence at which damage was first detected. For the sapphire substrate case, both the ar coating of $TiO_2/Al_2O_3/MgF_2$ and the bare substrate were found to exhibit high surface-damage thresholds, i.e., 28 to 54 J/cm^2. As anticipated, the exit surfaces were damaged at thresholds well below those of the entrance surface. The damage threshold of the ZnS/ThF_4 ar coating (17 J/cm^2) was found to be the lowest of all the sapphire samples tested.

Damage tests on CaF_2 substrates showed that commercially available ar coatings have damage resistance equal to that of the uncoated CaF_2 surface (Table I). The carbyne coatings on CaF_2 exhibited damage thresholds that varied over a wide range of fluences (14-25 J/cm^2). The best carbyne film was observed to have a damage resistance equal to that of the polished CaF_2 surface. On a microscopic scale, the coatings exhibited sample-to-sample variations as well as variations across a given sample. The damage levels could not be correlated with any particular deposition procedure. Figure 4 is a micrograph showing damage to a carbyne film deposited on CaF_2 and exposed at 23 J/cm^2 incident laser fluence. The damage is seen to have occurred preferentially at carbon-bearing spots in the carbyne film. It is likely that these carbon particles absorbed the laser radiation preferentially, but the regions remote from the damage spots were observed to be coated with a carbyne film that did not degrade at laser fluence levels at which the better commercially available films did degrade. Carbyne coatings of very high damage resistance should be achievable, therefore, if the coatings can be made free of particulate carbon matter.

A limited number of damage tests were performed on reflective optical components (Table I). An oxygen-free, high-conductivity (OFHC) copper mirror of "laboratory grade" surface finish was measured to have the highest damage threshold of all the component samples that were tested. Figure 5 is a micrograph of the laser damage sites on this polished mirror. The degree of damage is seen to be quite small, considering the high exposure fluence:

Fig. 4. Carbyne Film on CaF_2 Irradiated at 23 J/cm^2 Showing Localized Damage at Carbon-Bearing (black) Spots. Magnification 200X

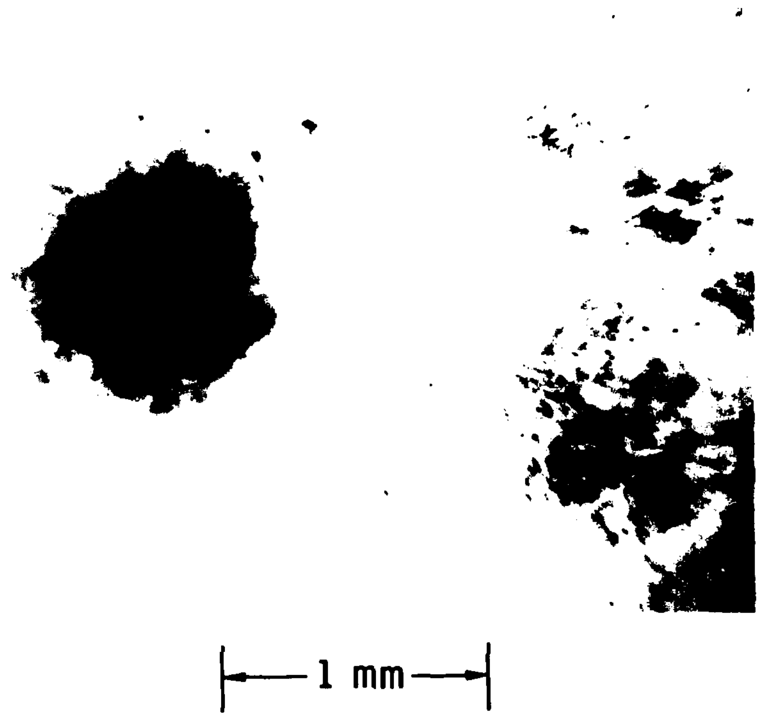

Fig. 5. Micrograph of Bare Copper (Spawr) Mirror Showing Damage Sites at Exposure Fluence of 58 J/cm^2 and DF-Laser Pulse Duration of 0.5 μsec (FWHM). Magnification 37.5X

58 J/cm^2. The carbyne-coated copper samples consistently failed at low fluences of 9.5 J/cm^2 or less. During the coating of copper samples, one half of each substrate was masked from the hot carbon source. Figure 6a gives clear evidence that particulate carbon contamination occurred on the half of the copper mirror that was considered to have been shielded from the heated carbon source. A method for interception of these carbon particulates must be devised for future coating studies. The irradiation of the carbyne vapor stream by a high-power laser beam would be one technique for interception of particulate material before its impingement on a substrate surface. Figure 6b is a micrograph showing laser damage to a carbyne film deposited on half of a polished copper substrate. Preferential damage at carbon-bearing sites is seen to be the failure mechanism, as was observed for the case of carbyne coatings on CaF_2 samples. We speculate that carbyne films on reflective optics would possess high damage resistance in the absence of these particulate carbon sites.

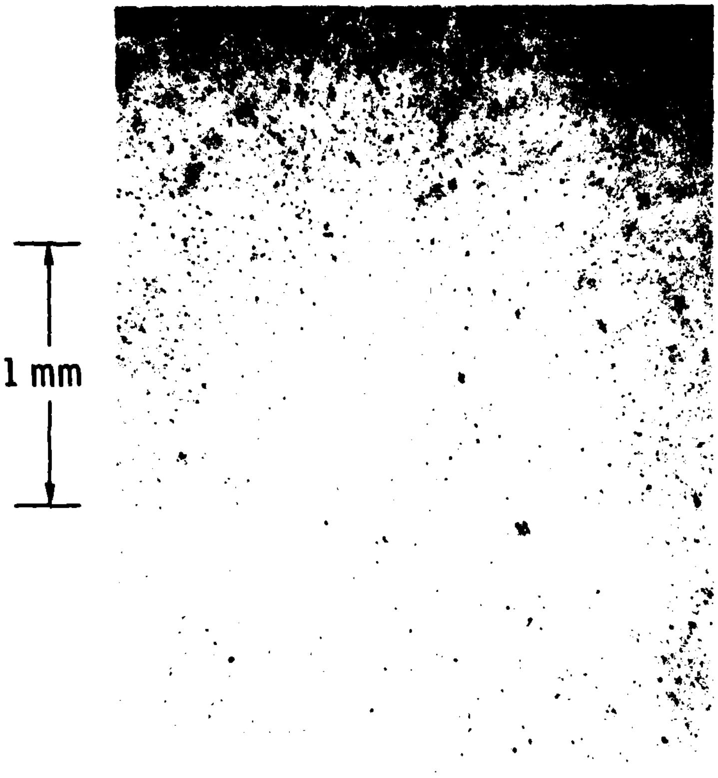

Fig. 6. Micrographs of Copper Mirror Showing Substrate Regions Masked and Unmasked from Carbyne Vapor Stream. Localized laser damage to carbyne coating occurs at carbon particulate sites. a. Masked Mirror Segment. No exposure to laser radiation. (Original magnification 37.5X) Carbon particulates have contaminated this copper substrate despite attempts to shield mirror segment from the carbyne effluent.

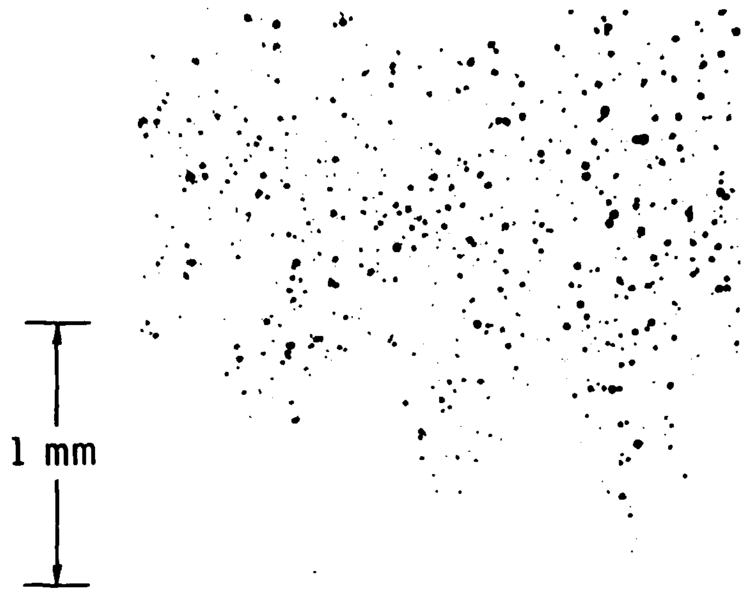

Fig. 6b. Unmasked Mirror Segment. Lower half not exposed to laser radiation. Upper half exposed to 9.5 J/cm^2. (Original magnification 37.5X)

IV. CONCLUDING REMARKS

Large-spot DF-laser damage thresholds have been investigated for bare substrates, commercially coated components, and hard carbyne films deposited on transparent and reflective optics. Damage resistance measurements on candidate DF-laser window components have shown that high-quality antireflection coatings are presently available for CaF_2 and Al_2O_3 substrates. Damage resistance of OFHC polished-copper mirrors has been excellent at DF chain-laser wavelengths. Unfortunately, the ability of these components to withstand high radiation fluxes in the presence of the hot corrosive gas flows of repetitively-pulsed systems is limited. Because of this limitation, we examined Aerospace carbyne coatings as candidate protective films for pulsed DF optical components. In our study, the best carbyne coatings survived high laser fluences of 25 J/cm^2. However, carbyne films as currently prepared for laser-damage evaluation generally showed numerous particulate carbon sites that were easily damaged. Areas free of these defects possessed high laser damage resistance. We recommend, therefore, that improvements in carbyne-coating preparation be pursued as a step toward the ultimate development of practical, damage-resistant films for use both in repetitively-pulsed DF laser devices and in other optical systems where corrosion resistance and high laser-damage resistance are essential characteristics.

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U.S. GOVERNMENT PRINTING OFFICE: 1984-783-004/2119

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